# Characterization of Coating for Replication

## Final Report
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**Objectives in Scope of Work:**

1) Study coating materials to be used to overcoat the nickel coated aluminum replication mandrels.

2) Study hardness and polishing procedures of coatings to determine which material is the best candidate.

3) Select a deposition procedure to make the coatings.

4) Characterize the deposition process to determine what parameters produce a hard durable coating to overcoat the replication mandrels.

5) Reports - Quarterly and Final

## 1. SCOPE AND PURPOSE

A process had been developed to create a lightweight telescope made of nested shells of Wolter-I type cylindrical telescopes. In the past, this type of telescope was large and heavy. The new process creates lightweight shells by replication from a mandrel. An aluminum mandrel is diamond turned to the correct shape, electroplated with nickel, diamond turned again, and polished to a very low surface roughness. Next gold is deposited and finally the piece is electroplated with stress-free nickel. The assembly is submerged in liquid nitrogen. The shell with a gold inner surface and stress free nickel outside surface separates from the mandrel due to the different thermal expansion coefficients. The inner gold surface has the same surface quality as the nickel coating onto which it was deposited.

The goal of this research was to study materials that could be coated over the electroplated nickel surface. Nickel is very soft and needs to be repolished after every replication. This decreases the life of the mandrel since the nickel surface can be

repolished a limited number of times before polishing breaks through to the aluminum mandrel surface. A hard durable material coated over the nickel surface would make the replication process much more efficient. A material that replicates the nickel surface or is polishable down to the desired surface roughness is ideal. Also this material must be hard enough that once the correct surface finish is reached, the surface doesn't degrade after many replications.

During this six month contract, the first three months of this effort focused on selecting the material to be deposited as the overcoat for the nickel coated aluminum mandrel. A number of materials were studied to determine which was the most durable and hard surface. Also, different approaches were studied to polish the different materials. In the first phase of this effort we selected TiN as the candidate material. It was the hardest and most durable material and showed the most promise during the polishing study.

The last three months focused on selecting a deposition process to produce TiN coatings in-house. Two approaches were available. First we considered rf- or dc-sputtering. The other option was ion-beam sputtering. Both efforts were pursued in parallel. Unfortunately, the rf/dc-sputtering chamber needed a number of hardware changes to correctly control the deposition process. The chamber had not been used in a number of years and a person experienced in using the chamber was not available to us. Also, many of the manuals were missing. This resulted in delays caused by trial and error of many of the supporting systems such as correct water flow, electrical diagnostic interlock, etc. In the end, the ion beam sputtering system was chosen for the TiN deposition because experience of other vendors (General Optics, Ion Tech) showed that TiN could be deposited without heating the substrates and the surface roughness was close to the values we desired.

## 2. SELECTION OF COATING MATERIAL

The selection of the coating material that was chosen to overcoat the replication mandrels is described in detail in the quarterly report. A coating material is wanted that is hard and durable and polishable down to < 5 angstroms. Summarizing the first three months of this effort , TiN was selected as the material to overcoat the replication mandrels. Stainless steel substrates were coated by Balzer's Tool Company. There were fifteen samples coated with titanium nitride (TiN), titanium carbonitride (TiCN), chromium carbide (CrC) and chromium nitride (CrN). The coatings were 3-4 μm thick for the standard coating runs and ~2 μm thick for the TiN mirror block run. Studies were made using a WYKO microscope to determine surface finish. A Nomarski phase contrast microscope was used to look at the structure of the films.

Initial studies show that TiN can be polished using SiC sandpaper. We brought the surface roughness from ~50 angstroms down to 14 - 15 angstroms. CrN is also a

candidate material that is easily polishable with $Al_2O_3$. We brought the surface roughness from ~ 250 angstroms down to 44 angstroms angstroms. We studied the structure of the film materials using a Nomarski phase contrast microscope. We found that TiN and CrN (as well as TiCN) have less surface structure than CrC. This would lead us to believe that these materials may be candidates for the overcoat onto the nickel. The mirror block of the TiN has dramatically less structure than the TiN of the standard block. Therefore, deposition conditions do effect the surface quality of the films. Finally, we have documented the procedure to polish stainless steel substrates to a surface roughness of 7-10 angstroms (Appendix B).

Unfortunately, many of the processes used for depositing TiN require the substrates be heated to very high temperatures. We are not able to heat the nickel coated aluminum mandrels because the different thermal expansion coefficients cause the nickel to separate from the aluminum. For our the initial studies described above and in more detail in the quarterly report, we looked at stainless steel samples coated by one of these processes. Another effect of heating the substrate is that the surface roughness of the coating will be crystalline in nature. Therefore, if the material made from a deposition with heating exhibits the qualities we need, then a different process that does not heat the substrate is likely to have a smoother surface finish.

## 3. SELECTION OF DEPOSITION TECHNIQUE TO DEPOSIT TIN

Selection of a deposition process to produce TiN coatings in-house was the focus in the last half of the contract. We had two options - rf/dc sputtering or ion beam sputtering. Both efforts were pursued. Unfortunately, the rf/dc-sputtering chamber needed a number of hardware changes to correctly control the deposition process. The chamber had not been used in a number of years and a person experienced in using the chamber was not available to us. Also, many of the manuals were missing. This resulted in delays caused by trial and error of many of the supporting systems such as correct water flow, electrical diagnostic interlock, etc. In the end, the ion beam sputtering system was chosen for the TiN deposition because: 1) the experience of other vendors (General Optics, Ion Tech) showed that TiN could be deposited without heating the substrates and the surface roughness was close to the values we desired, and 2) the investigator had begun modifying the ion beam sputtering chamber under another contract and was more familiar with the operation of the chamber. A TiN sample was provided by Ken Scribner of General Optics. The surface finish measurements were 2 - 3 angstroms. The deposition process used was ion beam sputtering with low deposition rates and no heating.

The ion-beam sputtering chamber had been assembled as a part of graduate work of the investigator. The vacuum chamber had previously been extensively modified. The components for ion-beam sputtering had been installed, but prior to this contract had not operated correctly, the bugs still needed to be worked out.

Because the investigator was more familiar with the ion-beam sputtering process and had modified the chamber herself, she pursued this approach, optimistic that results could be obtained before the contract ended. Also, at a conference a vendor (Ken Scribner of General Optics) said that his company was depositing TiN using ion-beam sputtering. Deposition rates are very slow, thus, the surface finish is very good . Also, the process does not require heat.

## 4. DESCRIPTION OF ION BEAM SPUTTERING

Ion beam sputtering utilizes a hollow cathode and a hollow cathode neutralizer. The hollow cathode is a plasma electron source. The hollow cathode assembly includes a hollow cathode tip with electron emissive insert, tip heater and keeper electrode all mounted on an alumina insulator disk. Argon or xenon gas is introduced through the hollow cathode tip where a plasma discharge is initiated by heating the cathode tip to about 1000 degrees C and applying 500 volts to the keeper electrode. The keeper power supply is designed so that this voltage drops rapidly to less than 50 volts when the keeper discharge starts. After a discharge has been established to the keeper, additional electrons can be drawn from the hollow cathode plasma to initiate and sustain the ion source discharge.

The hollow cathode baffle partially isolates the keeper discharge from the source discharge chamber. As a result, there is a potential difference across the baffle aperture of several tens of volts. As electrons pass through this aperture into the source discharge chamber they are accelerated by the potential difference into the energy range where the ionization cross section is near maximum. Some of the ions that are produced in the discharge reach the two grids and are focused by the positive screen grid and accelerated through the apertures in the negative accelerator grid. The accelerated ions form the directed beam of energetic ions.

The hollow cathode neutralizer then provides electrons to the positive ion beam. The neutralizing electrons are readily distributed within the conduction plasma of the beam to give a near uniform potential for most operating conditions.

## 5. STATUS OF DEPOSITING TIN USING ION BEAM SPUTTERING

Figure 1 shows a schematic of the ion beam sputtering process. The hollow cathode ion gun provides positive argon ions. The neutralizer emits electrons to neutralize the argon beam. The momentum of the argon atoms hitting the target causes titanium atoms to be released. At the same time, nitrogen is introduced into the plasma and a TiN film forms on the substrate.

The coating process for a single element compared to a compound coating is much simpler. For example, for a titanium coating, regardless of the distribution of the

Ti from the target, the substrate can be placed anywhere. This is because we can adjust the time of the deposition to get a desired thickness.

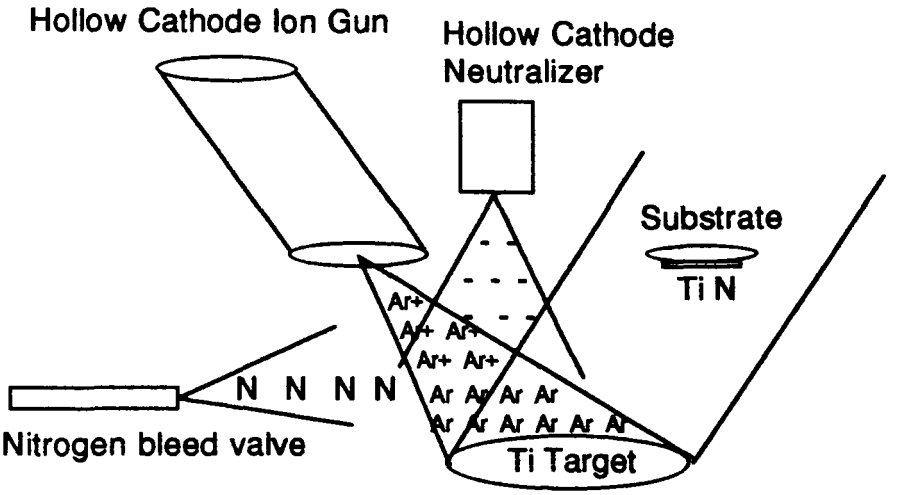


Figure 1. Schematic of ion sputtering process

The distribution of the material leaving the target in the deposition is very important in a compound coating. I found that to the left of the ion gun the coatings were mainly titanium. To the right of the gun, there was more of a mix of titanium and nitrogen (i. e. the coatings had more of a gold color), and to the extreme right the coatings were dark brown which is a sign of too much nitrogen. Ideally, the distribution of nitrogen throughout the chamber should be uniform.

I had positioned the substrate to the left of the ion gun assuming that the direction of the target atoms would follow the law of reflection. However, regardless of the amount of nitrogen, the coatings were never gold colored. However, I noticed that to the right of the gun the walls of the chamber were gold. Finally, when I removed the neutralizer from the chamber it was located to the extreme right of the gun. The shield on the neutralizer was a dark brown indicating an excess of nitrogen.

Thicknesses of the coatings were determined by running a deposition for a given amount of time and measuring the thickness on a Talystep profilometer. The Talystep uses a diamond stylus that detects small changes in height. Unfortunately, when the deposition was run for the same amount of time for a number of runs, the thicknesses were very different. I attributed this to problems with the hollow cathode neutralizer. It would initially operate normally, but shortly after the run started, the neutralizer would emit or not emit electrons to neutralize the ion beam. Thus,

thicknesses were not repeatable.

The hollow cathode neutralizer was acting erratically during all coating runs. When Ion Tech was consulted about the problem, they said many customers have difficulties with neutralizers and that the neutralizer needs to couple to the ion beam to operate reliably. However, there was nothing about the position of the neutralizer with respect to the ion gun that would lead them to believe that there was a problem. Late in the contract, I began to get a rare error on the neutralizer power supply. Ion Tech suggested a procedure to fix the problem. After following their procedure, the neutralizer was much more reliable. However, because the neutralizer was fixed so late in the contract, no coating characterization could be conducted. All of the information about coating thickness versus time and the correct amount of nitrogen to get gold colored films are no longer valid. This data must be collected again with a properly functioning neutralizer to get repeatable results.

Roughness measurements were never conducted on TiN coatings deposited at MSFC. This is because all coatings were deposited on microscope slides or float glass which typically have a surface roughness of ~ 30 angstroms. The coatings that we are trying to achieve should have a surface roughness of less than 5 angstroms. Alternately, the coatings should not increase the surface roughness of a substrate with a surface finish of less than 5 angstroms. Because the deposition process was not repeatable due to the neutralizer problems, the good Zerodur substrates with a surface finish < 10 angstroms were never used. I felt the super polished substrates were too valuable to use in a deposition process that was so unreliable. Once we determine the times necessary to deposit various thicknesses and the amount of nitrogen to get gold colored coatings with the neutralizer operating correctly, the valuable substrates can be used to determine the best coating parameters to achieve coatings with the smoothest finish.

## 6. CONCLUSIONS

TiN was chosen for the coating material to overcoat the replication mandrels. SiC was determined to be the best material to polish the TiN coatings. Ion beam sputtering was chosen as the deposition process. Heating is not required in the process and the coating can be deposited slowly leading to a better surface finish. Unfortunately, a repeatable process was not established due to equipment difficulties. However, now that the equipment is operating correctly, a repeatable process that creates a hard durable coating with a very good surface finish is realizable.

Darell Engelhaupt used TiN samples (provided by Balzers that we polished to achieve a surface roughness of ~ 14 angstroms) as mandrels for electroforming nickel disks. He proved that the TiN is a good replication surface. He was able to replicate from the mandrel 15 times with no noticeable change in surface finish of the TiN surface or the replicated nickel surface.

## 7. FUTURE WORK TO ESTABLISH REPEATABLE TIN COATING PROCESS

Now that the ion beam sputtering system is operating correctly with repeatable parameters, new runs must be made to determine the time required to deposit a TiN film with a desired thickness. Also, runs at different substrate positions must be made to determine the distribution of nitrogen throughout the chamber. Once the details of thickness and amount of nitrogen are selected, substrates with a surface roughness < 5 angstroms should be used to determine the surface finish. Deposition parameters can be optimized to decrease the surface roughness. Finally, testing should be conducted to ensure the coatings are hard and durable as expected.

The next step is to deposit TiN on nickel coated aluminum substrates. These samples must be tested to determine surface finish (does the sample need to be polished?), hardness and porosity. Replicating from these samples must be studied to see how gold deposited onto the TiN replicates the surface. Tests should be conducted to determine the number of replications possible before the TiN surface degrades.

Appendix A

## Procedure for MSFC Bldg. 4487 C-180 Ion Beam Sputtering Vacuum Chamber

### Pump down:

1. Turn on air - 60 psi is required (to allow roughing, foreline and diffusion valves to open and close).
   -in back of chamber - second valve from the left
2. Turn on nitrogen gas (to backfill chamber and blow dust off substrates).
   -in back of chamber - first valve from the left.
3. Turn on mechanical pump power on the bottom of the main console.
4. Open main water line
   -in back of chamber - blue faucet on floor
5. Open water line to diffusion pump
   -open left front door below chamber - third orange lever on right
6. Open foreline valve (foreline valve must remain open when diffusion pump is on
7. Turn on power to diffusion pump on bottom of main console and hit START button on box on left front door below chamber. After a few minutes check that diffusion pump is heating (red glow can be seen through hole in bottom of the diffusion pump). Allow the diffusion pump to heat up for 1 hour.
8. Turn on power to gas flow controller (GFC-1000) on main console - must warm up for at least 20 minutes
9. Open argon (high purity) and nitrogen (high purity) tanks that supply for ion sputtering process - tanks directly behind chamber ~30 psi each
10. Vent or backfill chamber with nitrogen
11. Load substrates and close chamber
12. Close foreline valve and open roughing valve
13. Alternately, open and close roughing and foreline valves to bring chamber pressure down to $< 10^{-1}$ torr while not letting the foreline pressure go above $10^{-1}$ torr. Once chamber pressure is below $10^{-1}$ torr, both the roughing and foreline valves can be open at the same time. **BEWARE** - Never open roughing and diffusion pump valves at the same time - oil may backstream into chamber
14. 10 minutes before diffusion pump is ready, fill cold trap with liquid nitrogen - fill cold trap every 1 1/2 hours
15. When diffusion pump is ready (pressure in chamber must be $<10^{-1}$ torr), close roughing valve, foreline must remain open
16. When pressure is $<10^{-3}$ torr, turn on power to ion gauge
17. Turn on ion gauge filament. Adjust control panel to correct pressure reading
18. It takes ~ 30 minutes to pump down to $7x10^{-6}$ torr

### Ion Sputtering Procedure:

1. Turn on water to target - open left front door to chamber, turn both top left and right

levers counterclockwise - send and return for water to target
2. Turn on gas one to hollow cathode neutralizer HCN(4.3 sccm) and gas two to hollow cathode HC (4 sccm). Wait 1 - 3 minutes to ensure that no oxygen is in HC tip
3. Turn on power supply MPS-3000 to ion gun and neutralizer
4. Check that settings on panel are similar to the data sheet provided by Ion Tech
5. Push SOURCE button
-the HC and HCN heater current should be increasing from ~2A to 7.25A and the voltage should be 511V
6. The heater current will get to 7.25A and the discharge current will start marked by a slow decrease in heater current to 3.75 A and a fast decrease in keeper voltage to 8-12 V
7. Let the source warm up for a few minutes after the heater current reaches 3.75A
8. Press the Beam button - this will start the sputtering process - nitrogen gas begins to fill the chamber when the beam button is pushed- check the data sheet to ensure that the values are in operating limits
9. Let the process continue for the length of time to achieve the desired thickness
10. Push the beam button again to stop the sputtering process
11. Push the source button to turn the source off
12. Turn the power off
13. Let the gas to the HC (gas two) and HCN (gas one) remain on for 1 hour before venting the chamber or starting the shut down procedure to leave the chamber under vacuum
14. Turn off HC and HCN gas on GFC-1000, turn off power to GFC-1000
15. Turn off water to target
16. Close argon and nitrogen gas tanks

**Shut down:**

1. Turn filament gauge range down to $10^{-4}$ torr and turn filament off
2. Close diffusion pump isolation valve, foreline must remain open
3. Turn off power to diffusion pump and hit red stop button on front door under chamber
4. Let diffusion pump cool down for one hour with foreline valve open and mechanical pump on
5. When you can touch bottom of diffusion pump below water lines, it is cool enough the shut down
6. Close foreline valve
7. Turn off water to diffusion pump and main water line
8. Turn of mechanical pump
9. Close air and nitrogen lines

Appendix B

**Process for polishing stainless steel substrates**

1. Grind with 30μm grit until all parts are cleared in and even - 50 lbs pressure
2. Machine settings:
   a. Spindle - 34
   b. Eccentric - 21
   c. Off-center-inches - 0
   d. Travel-inches - 2
3. Grind with 30μm grit for one half hour - 50 lbs pressure
4. Grind with 20μm grit for one half hour - 50 lbs pressure
5. Grind with 9μm grit for one half hour - 38 lbs pressure
6. Grind with 5μm grit for one half hour - 30 - 35 lbs pressure
7. Polish with 0.5μm diamond on Pellon pad until the measured surface roughness reaches ~ 7 angstroms